IS 6962 (1991): 3,9-Dibromobenzanthrone, technical-Specification [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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भारतीय मानक

3, 9-डाइब्रोमोवेंजानथ्रोन, तकनीकी — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

3, 9-DIBROMOBENZANTHRONE, TECHNICAL — SPECIFICATION

(First Revision)

UDC 667·282·41

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

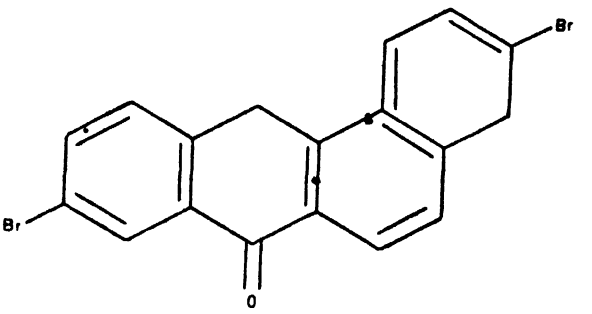
December 1991

Price Group 3

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

3, 9-Dibromobenzanthrone is an important intermediate used in the manufacture of vat dyes. Its molecular, structural formula and molecular mass are given below:

Molecular Formula	Structural Formula	Molecular Mass
$C_{17}H_8OBr_2$		388·0

3, 9 Dibromobenzanthrone
C. A. S. Registry Number [81—98—1]

This standard was first issued in 1973. The committee responsible for the preparation of this standard decided to revise it in order to update the same. In the present version (*first revision*), the requirement of boromine content has been deleted since the same was not found to be relevent in respect of its quality but requirement of assay has been introduced. Besides, requirements of 3-bromobenzanthrone overbrominated product as tribromobenzanthrone and matter insoluble in xylene have also been incorporated.

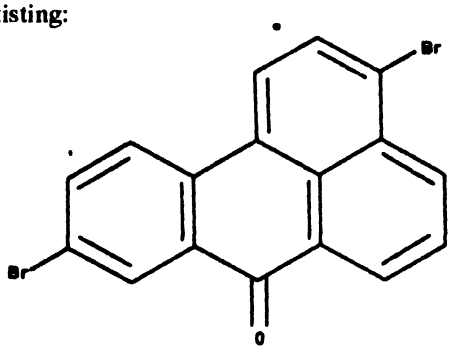
For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 1 JUNE 1993
TO
IS 6962 : 1991 3, 9-DIBROMOBENZANTHRONE, TECHNICAL — SPECIFICATION

(First Revision)

(*Forword, second para, molecular formula and structural formula*) — Substitute the following for existing:

$C_{17}H_8Br_2O$



3, 9 Dibromobenzanthrone
C. A. S. Registry Number [81—98—1]

(*Foreword, third para, line* 3) — Read 'bromine' *for* 'boromine'.

(*Page* 1, *clause* 2.1, IS 1070 : 1977, *title*) — Delete 'Specification for' and read 'Water' for 'water'.

[*Page* 1, *Table* 1, *Sl No* (i), *col* (4)] — Read 'A-2' *for* 'A-3'.

(*Page* 3, *clause* A-5.6.2, *line* 9 *and Note* 1, *line* 2) — Read 'nm' *for* 'mm'

(*Page* 4, *clause* A-5.7.1, *title*) — Read '*3 bromobenzanthrone*' *for* '*3, 9-bromobenzanthrone*'.

(*Page* 4, *clause* A-5.7.1, *lines* 16 *and* 20) — Read 'quantitatively' *for* 'qualitatively'.

(*Page* 4, *clause* A-5.7.1, *line* 23) — Read '393.5' *for* '398.5'.

(*Page* 4, *clause* A-5.7.1, *line* 29) — Read 'obtained' *for* 'contained'.

(*Page* 4, *clause* A-5.7.2, *line* 7) — Delete 'mass'.

(*Page* 5, *clause* A-6, title) — Add 'INSOLUBLE' between 'MATTER' and 'IN'.

Indian Standard

3, 9-DIBROMOBENZANTHRONE, TECHNICAL — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 3, 9-Dibromobenzanthrone, technical.

2 REFERENCES

2.1 The following Indian Standards have been referred to:

IS No.	*Title*
460 (Part 1) : 1985	Wire cloth test sieves (*third revision*)
1070 : 1977	Specification for water for general laboratory use (*second revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) (*third revision*)
5299 : 1969	Methods of sampling and tests for dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be in the form of greenish yellow powder and shall be free from visible impurities.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be suitably packed in steel drums (*see* IS 2552 : 1989) lined with polyethylene or as agreed to between the purchaser and the supplier.

4.2 Marking

The container shall be marked with the following :

a) Name of the material;

b) Tare, gross and net mass of the material;

c) Indication of the source of manufacture;

d) Batch number; and

e) Month and year of manufacture.

4.2.1 The containers may also be marked with the Standard Mark.

5 SAMPLING

5.1 The material shall be sampled in accordance with 3 of IS 5299 : 1969.

Table 1 Requirements for 3, 9-Dibromobenzanthrone, Technical

(*Clause* 3.2)

Sl No.	Characteristic	Requirements	Method of Test, Ref to Annex A
(1)	(2)	(3)	(4)
i)	Melting range	Shall melt within the range of 3·0°C including 236·0°C	A-3
ii)	Moisture content, percent by mass, *Max*	0·5	A-3
iii)	Sulphated ash, percent by mass, *Max*	1·0	A-4
iv)	Assay (on dry basis), percent by mass, *Min*	85·0	A-5
v)	3-bromobenzanthrone, percent by mass (on dry basis), *Max*	5·0	A-5
vi)	Over brominated product as tribromobenzanthrone, percent by mass (on dry basis), *Max*	5·0	A-5
vii)	Matter insoluble in xylene, percent by mass, *Max*	1·0	A-6

5.2 Number of Tests

5.2.1 Test for the determination of melting range and moisture shall be conducted on each of the individual samples.

5.2.2 Test for the determination of remaining characteristics, namely, description, sulphated ash, assay, 3-bromobenzanthrone, tribromobenzanthrone and matter insoluble in xylene shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirement of melting range and moisture if each of the individual test results (*see* **5.2.1**) satisfies the relevant requirement given in Table 1.

5.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics (*see* **5.2.2**) tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant requirements given in **3.1** and Table 1.

ANNEX A

(*Table* 1)

METHODS OF TEST FOR 3, 9-DIBROMOBENZANTHRONE, TECHNICAL

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure Chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF MELTING RANGE

A-2.1 Determine the melting range of the sample in accordance with the method given in **8** of IS 5299 : 1969.

A-3 DETERMINATION OF MOISTURE CONTENT

A-3.1 Moisture shall be determined by drying in an oven at 100° to 110°C in accordance with the method given in **9** of IS 5299 : 1969.

A-4 DETERMINATION OF SULPHATED ASH

A-4.1 Determine sulphated ash of the sample in accordance with the method given in **11.2** of IS 5299 : 1969.

A-5 ASSAY

A-5.1 Prepared Sample

Dry the material at 105°C to constant mass. Grind and mix well.

A-5.2 Outline of the Method

Purity of the material is estimated by chromatographic method. 3, 9-Dibromobenzanthrone is separated chromatographically and determined quantitatively by spectrophotometer.

A-5.3 Apparatus

A-5.3.1 *Chromatographic Column*

A glass tube of 35 cm long and having an internal diameter 2·5 cm fitted with a stopcock at the lower end. Set up the column vertically such that the percolation passing through the column can be collected in 250 ml conical flask. Place a cotton wool plug in a tube and press it to the bottom by means of a glass rod, flattened latitudinally at the end.

A-5.3.2 *Sintered Glass Crucible* — G 4.

A-5.3.3 *Ultraviolet Lamp*

A-5.3.4 *Spectrophotometer*

A-5.4 Reagents

A-5.4.1 *Alumina* — Aluminium oxide standardized for chromatographic adsorption analysis as below.

A-5.4.1.1 *Fineness*

	Percentage
Retained on 180 micron IS sieve	0·5 *Max*
Retained on 75 micron IS sieve	78 to 82
Retained on 53 micron IS sieve	88 to 92

A-5.4.1.2 *Activation*

Activate alumina by heating at 100°C for 1 hour. Allow it to cool. Use slightly warm alumina for column.

A-5.4.2 *Xylene*

Freshly distilled, dried over alumina and filtered.

A-5.5 Preparation of Standard 3, 9-Dibromobenzanthrone

Dissolve 400 mg of *prepared sample* (**A-5.1**), in 100 ml of xylene by refluxing for 20 minutes. Cool to room temperature and filter. Load 5 ml of filtered solution on the column, prepared with activated alumina and xylene as described in **A-5.6.1**. When the level of the liquid falls to the filter paper disc, wash down the sides of column with small lots of xylene so that all the coloured solution is washed into the column. Add xylene carefully to the top of the column so as not to disturb its surface. Continue to wash down the column with xylene to develope the chromatogram. When viewed under ultraviolet light, following bands are normally visible:

a) Pale yellow band of over brominated product at the bottom;
b) Yellow main band of 3, 9-dibromobenzanthrone;
c) Yellow band of 3-bromobenzanthrone; and
d) Strongly absorbed band at the top of the cloumn.

A-5.5.1 Flute a band of overbrominated product completely with xylene and discard. Now fill up the column with xylene and continue elution. Collect the main band of 3, 9-dibromobenzanthrone in a flask. A battery of several tubes is kept in operation for collecting a sizeable quantity of the material. Filter to remove any stray alumina particles. Concentrate the total quantity of eluate of 3, 9-dibromobenzanthrone by distillation of the eluent, until crystallisation occurs on cooling at room temperature. Filter the crystallised material through sintered glass crucible. Wash with small quantity of petroleum spirit. Dry at 100±5°C.

A-5.5.2 In order to check its purity, weigh accurately 40 mg of pure material and dissolve in 200 ml of xylene by refluxing for 20 minutes. Cool and transfer into 250-ml standard volumetric flask using xylene. Make up to mark and mix well. Call this solution *R*. Load 5·0 ml of solution *R* on a chromatographic column prepared with activated alumina and xylene as described in **A-5.6.1**. Collect the band completely (only one band should appear on the column) in 50 ml standard volumetric flask. Make up to mark with xylene and mix well. Determine its optical density at 398·5 nm in 1 cm silica cell specially matched on standard spectrophotometer.

Pipette another 5·0 ml of solution *R* in 50 ml standard volumetric flask and make up to mark with xylene. Mix well and determine its optical density in the same way. The difference in the two figures of optical density amounting to more than 0·003 indicates the presence of an impurity. The chromatographic purfication shall, in such a case, be repeated until this check test is satisfied.

A-5.6 Procedure

A-5.6.1 *Preparation of Chromatographic Tube*

Prepare a slurry of about 100 g of activated alumina in xylene and pour it into the tube. Stir slowly and continuously with the glass rod during the setting of the adsorbent, withdrawing the stirring rod gradually as the column builds up. (This stirring technique reduces 'coning' and gives better separation of the various components). Wash down the sides of the column with xylene. Allow the column to settle but not to run dry leaving about 1 cm of solvent above the top of the adsorbent. Add filter paper disc (cut to fit the diameter of the top of the tube) to the top of the column.

A-5.6.2 *Standard Calibration Graph*

Prepare a number of standard solutions of chromatographically pure 3, 9-dibromobenzanthrone as obtained in **A-5.5** in xylene varying in concentrations from 4 mg/250 ml to 20 mg/250 ml with a difference of 4 mg/250 ml between successive concentrations. Dilute 5 ml of each concentration to 25 ml with xylene. Take optical density readings of all the concentrations at wavelength 398·5 mm in specially matched 1 cm silica cells.

Plot the calibration curve of concentration against optical density.

NOTES

1 Maximum absorption has been observed at a wavelength 398·5 mm by standard instrument. However, this may need checking with the particular instrument to be used. Several readings for absorption using solutions of varying concentrations are taken at different wavelengths and the one for maximum absorption determined from the graph.

2 The standard calibration curve and the spectrophotometer shall be checked for accuracy periodically.

A-5.6.3 *Determination of Purity of Sample*

Weigh accurately 200 mg of *prepared sample* (A-5.1). Transfer quantitatively to a 500 ml erlenmayor flask using 200 ml of xylene. Reflux for 20 minutes using water condensor. Cool to room temperature by keeping in a water bath. Transfer quantitatively to dry, clean 250 ml standard volumetric flask. Dilute to mark with xylene. Mix well. Call this solution *Q*.

A-5.6.3.1 Prepare a xylene soaked alumina column using chromatographic tube 35 cm long with 2·5 cm internal diameter, and activated alumino as adsorbent with xylene as a solvent. Pack the column as in A-5.6.1.

A-5.6.3.2 Load 5·0 ml of solution *Q* by means of a pipette to the top of the column. When the level of the liquid falls to the filter paper disc, wash down the sides of the column with small lots of xylene so that all the coloured solution is washed into the column. Add xylene carefully to the top of the column so as not to disturb its surface and continue to wash down the column to develope the chromatogram.

When viewed under ultraviolet light, the following bands are normally visible:

a) Pale yellow band of overbrominated product at the bottom;
b) Yellow main band of 3, 9-dibromobenzanthrone;
c) Yellow band of 3-bromobenzanthrone; and
d) Strongly absorbed band at the top of the column.

Elute the bands of overbrominated product, 3,9-dibromobenzanthrone and 3-bromobenzanthrone in order collecting them separately in 100 ml. 250 ml and 250 ml standard volumetric flask respectively. Make the volume of the elutes to the mark with xylene. Mix well. Call them solutions *X*, Y, *Z* respectively.

Measure optical density of these solutions by means of spectrophotometer under the conditions given below, using xylene in the blank cell in each case.

Solution	*Component*	*Wavelength* nm	*Absorption Cell Length* (cm)
X	overbrominated product	398·5	4
Y	3, 9-dibromobenzanthrone	398·5	1
Z	3-bromobenzanthrone	393·5	1

A-5.7 Calculation

$$\text{Assay, percent by mass} = \frac{B \times 10 \times 100}{C}$$

where

B = mass obtained in g of 3, 9-dibromobenzanthrone in 25 ml solution, from calibration graph; and

C = mass in g of 3, 9-dibromobenzanthrone taken for test.

A-5.7.1 *Standard Calibration Graph 3, 9-bromobenzanthrone*

Prepare a number of standard solution of chromatographically pure 3-bromobenzanthrone in xylene, varying in concentration from 4 mg/250 ml to 20 mg/250 ml with difference of 4 mg/250 ml between successive concentrations. Dilute 5 ml of each concentration to 250 ml with xylene. Take optical density reading of all the concentrations at a wavelength 393·5 mm using specially matched 1·0 cm silica cells.

Plot the calibration curve of concentration against optical density.

Preparation of sample solution:

Weigh accurately 200 mg of prepared sample (A-5.1). Transfer qualitatively to a 500 ml erlenmayor flask using 200 ml of xylene, reflux for 20 minutes using water condensor. Cool to room temperature by keeping in a water bath. Transfer qualitatively to dry clean 250 ml standard volumetric flask. Dilute to mark with xylene. Mix well. Take optical density ready at a length 398·5 nm using specially watched 1·0 cm silica cells.

Calculations

$$\text{3-Bromobenzanthrone, percent by mass} = \frac{D \times 100}{E}$$

where

D = mass contained in g of 3-bromobenzanthrone in 250 ml solution, from calibration graph; and

E = mass in g of sample taken for test.

A-5.7.2 Overbrominated product a s tribromobenzanthrone

Optical density of 100 percent tribromobenzanthrone in 4 cm cell, keeping same concentration as sample = 5·0

$$\text{Overbrominated product as tribromobenzanthrone mass, percent by mass} = \frac{S \times 100}{5{\cdot}0}$$

where

S = optical density of overbrominated product in the sample under test, in 4·0 cm cell.

NOTE — The factor 5·0 has been determined for a particular instrument using 100 percent pure sample. But it is sufficiently accurate for the purpose of this test, for use with any other instrument.

A-6 DETERMINATION OF MATTER IN XYLENE

A-6.1 Procedure

Weigh accurately 200 mg of previously sieved and dried material. Transfer it to 500 ml erlenmayer flask using 250 ml of xylene and reflux for 15 minutes on electric hot plate. Filter hot through a tared sintered glass crucible (G 4) and wash the residue with xylene till filtrate is colourless. Dry the crucible along with the insoluble to constant mass at 100 ± 5°C.

A-6.2 Calculation

$$\text{Matter insoluble in xylene, percent by mass} = \frac{M_1 \times 100}{M}$$

where

M_1 = mass in g of insoluble; and

M = mass in g of material taken for test.

Standard Mark

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

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